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SPECIFICATIONS FOR CLEANING, FUSION WELDING, AND POSTHEATING TANTALUM AND COLUMBIUM ALLOYS

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This information is being published in preliminary form in order to expedite its early release.

ABSTRACT

This report consists of five NASA-Lewis Research Center specifications on the cleaning, fusion welding, and postheating of tantalum and columbium alloys. These specifications are intended for the use of NASA-Lewis and its contractors to help insure the production of defect-free weldments.

SUMMARY

Five specifications have been prepared on the subjects of cleaning, fusion welding, and postheating columbium and tantalum alloys. Adherence to these specifications should help assure weldment reliability. These specifications are intended to serve as standards for future programs conducted by NASA-Lewis Research Center and its contractors. In many areas, the specifications are stringent; however, the degree of care specified herein is believed to be justified because of material expense, high fabrication costs, and required service performance.

SPECIFICATIONS FOR CLEANING, FUSION WELDING, AND POSTHEATING TANTALUM AND COLUMBIUM ALLOYS

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INTRODUCTION

The group of specifications included herein cover the cleaning, fusion welding, and postheating of tantalum and columbium alloys. These specifications are intended to serve as standards for future programs conducted by NASA-Lewis Research Center and its contractors in fabricating aerospace test components from these alloys. They are included in this report format to document the current state-of-technology and for possible use by other organizations working with these alloys.

Requirements listed in these specifications are stringent in many areas. But this level of care is felt to be necessary in order to give maximum quality assurance. Tantalum and columbium alloys are extremely sensitive to pickup of impurities, so great care must be exercised to minimize the possibilities of contamination. Components fabricated from these alloys are extremely expensive because of material and processing costs. Service failure due to the use of improper procedures in the preparation of weldments would incur additional costs and lost service time. Thus, for advanced space power systems and other aerospace applications, columbium and tantalum alloy weldments must be highly reliable. Close adherence to these specifications should help assure such reliability.

The specifications are based on General Electric Company (Nuclear Systems Programs) and other documents resulting from the welding technology that was developed on NASA-Lewis sponsored contract programs (references 1, 2, 3, and 4). However, several important changes have been made and are incorporated in the specifications included in this report. These changes have resulted from recent experiences of several organizations working with these alloys.*

*Helpful comments on these specifications were received from W. R. Young of General Electric Company and G. G. Lessmann of Westinghouse Electric Company.

The specifications included herein are as follows:

Number	Title of Specification
RM-1	Chemical Cleaning of Columbium, Tantalum, and Their Alloys
RM-2	Gas Tungsten-Arc Welding of Columbium, Tantalum, and Their Alloys
RM-3	Electron Beam Welding of Columbium, Tantalum, and Their Alloys
RM-4	Resistance Spot Welding of Refractory Metal Foil to Refractory Metal Components
RM-5	Postheating of Cb-1Zr and T-111 (Ta-8W-2Hf) Weldments

NASA-LEWIS SPECIFICATION NO. RM-1

CHEMICAL CLEANING OF COLUMBIUM, TANTALUM, AND THEIR ALLOYS

June 1971

I. SCOPE

This specification establishes the requirements for the chemical cleaning of columbium, columbium alloy, tantalum, and tantalum alloy products. Chemical (acid) cleaning is not permissible on subassemblies that contain open crevices.

II. REQUIREMENTS

A. Materials

The materials required under this specification are hydrofluoric acid (48% solution), nitric acid (70% solution), sulfuric acid (95% solution), and flake sodium hydroxide. All concentrations are in percent by weight.

B. Equipment

1. Acid Tank: The acid tank must be resistant to hydrofluoric/nitric/sulfuric acid mixtures.

2. Wash Tank: The wash tank shall have controlled heat and agitation.

3. Fixtures: The product retaining fixtures shall be resistant to hydrofluoric/nitric/sulfuric acid mixtures.

4. Personnel Protection: Safety equipment and clothing shall be used.

5. Water Control: Water softener and deionizer equipment shall be provided.

C. Safety

The solutions required for the proper chemical cleaning of columbium and tantalum materials are EXCEEDINGLY HAZARDOUS. Extreme caution, as outlined below, shall be exercised in the preparation and use of the solutions:

1. Clothing: Protective clothing shall be mandatory.

2. Signs: Proper warning signs shall be posted at each entrance to the cleaning area.

3. Access: Access to the facility shall be to authorized personnel only.

4. Safety Center: An emergency safety center shall be established and equipped with the proper acid antidotes, equipment, and directions for their usage, as specified and demonstrated by the plant industrial hygienist.

D. Surface Preparation

All surfaces of the columbium, columbium alloy, tantalum, and tantalum alloy products shall be free of grit, metallic chips, grease, oil, fingerprints, or any organic markings. Solutions used for cleaning shall be of sufficient purity to prevent accumulation of undesirable residues after drying.

E. Final Water Rinse

The deionizer shall be capable of maintaining a specific resistance of the final rinse water of greater than 200,000 ohms/cm.

III. PROCEDURE

A. Solution

The composition of the solution shall be as follows:

	Columbium and Columbium Alloys (parts by volume)	Tantalum and Tantalum Alloys (parts by volume)
Hydrofluoric acid (48% solution by weight)	1.5	1
Nitric acid (70% solution by weight)	2	4
Sulfuric acid (95% solution by weight)	1	1
Water	5.5	2

The quantity of cleaning solution shall be calculated or measured so that only enough acid is prepared for the pickling operation. (Acids should always be added to the water; NEVER the reverse.) The prepared solution shall be thoroughly agitated to insure complete mixing. The temperature of the solution should not exceed 50^{o} C (125^{o} F) during pickling. The acid solution should be disposed of as soon as possible after it is no longer useful, as described in Section IV.A, by neutralizing with sodium hydroxide solution to a pH range of 5.2-10.8 in a sump tank. The neutralized solution will then be diluted by 50% with water before the sump is pumped out into the plant waste system.

B. Loading

The product-retaining fixture shall be loaded so that the cleaning solution shall have free access to all surfaces of the charge. To assure uniform cleaning, the retaining fixture should be loaded with products with as similar dimensions as possible.

C. Acid Immersion

The chemical cleaning operation shall be scheduled immediately prior to any fabrication process in order to maintain a minimum storage time for cleaned stock.

The products to be cleaned shall be totally immersed in the solution and agitated by gently raising and lowering the retaining fixture beneath the surface of the solution. If hollows (tubes, pipes, cylinders, etc.) are being cleaned, the agitation shall be achieved by moving the rack back and forth in the solution to insure a continuous supply of fresh solution to the internal surfaces. The immersion time will vary depending on the mass and configuration of the products. The amount of stock removal required shall be specified by the Project Manager.

D. Water Rinse

After the acid immersion, the retaining fixture shall be raised above the solution and immersed immediately in clean, flowing domestic water and agitated by air sparging. Fast transfer from acid solution to water rinse shall be used in order to prevent surface drying of the acid solution. Immersion in the water rinse shall be for no less than 30 minutes with the water at a temperature above 50° C (125° F). Any residue remaining on the surface of the cleaned-and-rinsed products is to be wiped off with a clean cotton cloth. Internal surfaces of hollows are also required to be wiped clean with a clean cloth by swabbing. After the cleaning of any surface with a cloth, the product is to be re-immersed in flowing water and agitated by a back-and-forth movement for a period of not less than five minutes. Note: Rinsing is a most important step because pickling residues can cause surface contamination or severe outgassing of the samples on heating. In edge preparation studies, weld porosity has been associated with pickling residue.

E. Deionized Water Rinse

Following the water rinse, the products shall be immersed for a minimum of five minutes in deionized water (sonic agitation if possible) at a temperature above 50° C (125° F). The resistance of the water is to be maintained above 200,000 ohms/cm. Distilled water meeting the resistance requirements may be substituted for deionized water. The deionized water rinse tank should not contain any visible deionizing resin.

F. Drying

Drying of the cleaned and rinsed products can be accomplished either by drip drying on the retainer fixture, by rewracking on a drying fixture, or by rinsing with ethyl alcohol followed by drip drying or hot air flash drying.

G. Vacuum Heat Treatment

At the discretion of the Project Manager the cleaned and dried products may be subject to vacuum heat treated at 1100° C (2012° F) for 15 minutes to decrease the possibility of hydrogen pickup from the prior cleaning operation.

H. Preparation for Delivery

The cleaned and dried products shall be transferred immediately to clean polyethylene or clean glass as the only acceptable container materials. Special care must be taken to avoid scratching of the polyethylene and thus contaminating the metal system. Cleaned products shall be handled using clean nylon gloves.

IV. QUALITY ASSURANCE PROVISIONS

A. Stock Removal Rate

The stock removal rates of columbium, columbium alloy, tantalum, or tantalum alloy products are to be determined experimentally a minimum of once a day during any period of time that the acid solution is in continuous use or prior to the cleaning of the products, in the event that the acid solution has not been used for a long period of time and significant evaporation has occurred. The stock removal rate is to be determined by immersing two tabs, approximately 1 mm x 1.3 cm x 1.3 cm (0.040-inch x 0.5-inch x 0.5-inch), of the material to be cleaned in the acid solution for a period of 10-minutes. The acid solution is to be replaced if the stock removal rate exceeds 0.08 mm (0.003-inch)/side or is less than 2.5 um (0.0001-inch)/side.

B. Chemical Analysis

At intervals of at least once a month, chemical analysis of a tab (described in paragraph IV(A)) shall be performed for hydrogen. A hydrogen increase of greater than 2 ppm over the original product analyses shall be cause for replacing the acid or adjusting the composition to reduce the extent of hydrogen pickup.

C. Visual Examination

Visual examination of the cleaned products after the final rinse shall reveal a continuous film of water over the surfaces. A discontinuous film of water on the surfaces is indicative of improper or insufficient cleaning. Any product that shows a discontinuous film of water on its surface shall be recycled through the acid cleaning solution.

D. Rejection

Weldments and components not cleaned according to this specification shall be rejected.

E. Reports

A report shall be submitted with the cleaned weldments or components. The information listed in Exhibit I shall be included, but not necessarily in that format.

EXHIBIT I

PROCESS CONTROL RECORD

Date	Subject
Contract No.	Chemical Cleaning of Columbium, Tantalum, and Their Alloys

Part Name and Material:

Amount of Stock Removed:

Process Control Procedure:

General Observations:

NASA-LEWIS SPECIFICATION NO. RM-2

GAS TUNGSTEN-ARC WELDING OF

COLUMBIUM, TANTALUM, AND THEIR ALLOYS

JUNE 1971

I. SCOPE

This specification establishes the procedures, process substantiation, and quality requirements for gas tungsten-arc welding columbium, tantalum, and their alloys. Field welding as defined in Item III is also covered.

II. APPLICABLE DOCUMENTS

AWS A3.0-69	Terms and Definitions
AWS A2.0-68	Standard Welding Symbols
AWS A5.12-69	Tungsten Arc-Welding Electrodes
NASA-Lewis Specification No. RM-1	Chemical Cleaning of Columbium, Tantalum, and Their Alloys
NASA-Lewis Specification No. RM-5	Postheating Cb-1Zr and T-111 (Ta-8W-2Hf) Weldments
AWS A2.2-69	Nondestructive Testing Symbols
ASTM E94-62T	Tentative Recommended Practice for Radiographic Testing
ASTM E142-64	Controlling Quality of Radiographic Testing
ASTM E165-65	Liquid Penetrant Inspection

III. DEFINITIONS

A. Gas Tungsten-Arc Welding

An arc-welding process wherein coalescence is produced by heating with an arc between a single tungsten (noncomsumable) electrode and the work. Shielding is obtained from argon or helium. A filler metal may or may not be used. (This process has sometimes been called TIG welding.)

B. Field Welding

Gas tungsten-arc welding components in vacuum-purge chambers at the facility site: for components that are too large to assemble in available vacuum-purge welding chambers designed for refractory metal welding.

IV. REQUIREMENTS

A. Materials

1. Inert Gases: Considering normal weld chamber atmosphere degradation rates and the period of time involved in a typical welding cycle, ultra-pure helium or argon is required. Impurities in the inert gases shall be less than 2 ppm oxygen, less than 7 ppm nitrogen, and less than 2 ppm water vapor.

2. Electrodes: The tungsten electrodes shall conform to Class EWTh-2 per AWS 5.12-69.

3. Filler Wire: Welding filler material composition shall conform to the chemical requirements for the base metal, unless otherwise specified.

B. Equipment

1. Welding Machine: A direct current, arc-welding machine shall be used.

2. Welding Chamber:

   a. Chambers Designed for Refractory Metal Welding - The welding shall be done in an enclosed chamber that can be evacuated to less than 1×10^{-5} torr. The leak rate shall result in a pressure increase of not more than five microns/hour starting at 1×10^{-3} torr pressure or less. The chamber shall be equipped with glove ports.

   b. Field Welding Chambers - Field welding shall be done in an enclosed leak-tight chamber that can be evacuated to less than 25 microns pressure. The chamber shall be constructed of stainless steel with Pyrex (or equivalent heat-resistant glass) view ports. The chamber shall be equipped with glove ports. Chamber seals in contact with the refractory metal components shall be an adequate distance from the weld zone to prevent overheating and contamination of the work.

3. Gloves: Neoprene rubber gloves, 0.08-mm (0.030-inch) thick, shall be sealed gas-tight to the glove ports. The gloves shall be sulfur-free type and shall be qualified per paragraph V.B.2.

4. Welding Torch: The welding torch or head shall be gas or water cooled. The water passage of a water-cooled torch or head shall be permanently sealed. Cable and hose connections shall be vacuum tight. Exposed metal parts of the welding torch head shall be made of refractory metal or covered by refractory metal. For field welding inert gas shall be circulated through the torch gas hose during chamber purging and welding.

C. Cleaning and Handling

1. Components: The filler wire and parts to be joined shall be chemically cleaned in accordance with NASA-Lewis Specification No. RM-1, "Chemical Cleaning of Columbium, Tantalum, and Their Alloys."

2. Assemblies: If intermediate processing has not maintained the cleanliness, the metal adjacent (nominally 10-cm (4-inches) on each side of the joint) to the weld shall be recleaned per NASA-Lewis Specification No. RM-1.

3. Handling: Cleaned parts, components, and assemblies shall be handled with clean, lint-free gloves.

4. Metal Vapors: Deposits of metal vapors produced by the welding process shall not be considered detrimental or subject to cleanliness requirements. These films, however, may be removed by wire brushing with refractory metal brushes. Steel or other nonrefractory metal brushes must not be used.

D. Joint Preparation

1. Design: The edges of the parts shall be prepared for welding by machining or filing as shown on the engineering drawing.

2. Fixturing: The joints to be welded shall be positioned to provide proper alignment, match of parts, and root opening. The joint edges shall not be misaligned more than 20 percent of the thinner section being joined or 1.6-mm (1/16-inch), whichever is less.

3. Fixture Materials: The portion of the fixture that contacts refractory metal alloy components shall be made of molybdenum, tungsten, columbium, tantalum, or their alloys. Under no

circumstances shall any part of the refractory metal alloy component contact any nonrefractory metal during welding.

4. Fixture Cleanliness: The fixture shall be clean and free of surface contamination.

5. Tack Welds: After the parts are properly positioned, tack welds may be used to maintain alignment during welding. Tack welds shall have complete fusion and penetration to the weld-joint root.

E. Welding Procedures

1. Qualification: Welding shall be done using equipment and materials which have been qualified per paragraph V.B.

2. Preparation of Chamber Atmosphere

   a. Chambers Designed for Refractory Metal Welding - Prior to each welding cycle, the welding chamber shall be evacuated to 1×10^{-4} torr or better and the fixturing heated to at least 50° C (120° F) for a minimum of four hours. After cooling, the vacuum shall be 1×10^{-5} torr or better before filling with inert gas containing less than 2 ppm each of oxygen and water vapor by volume. Before removing the glove-port cover plates, the evacuation line to the glove ports shall be closed.

   b. Field Welding Chambers - After evacuation to less than 25 microns pressure, the chamber shall be filled with inert gas. (Experience has shown that it is desirable to maintain the 25 micron pressure level for a period of at least eight hours prior to filling with inert gas.) Inert gas flow through the chamber at a rate between 0.84 and 2.8 std. cu. meters (30 and 100 std. cu. ft.) per hour (depending on a chamber volume) shall then be established and maintained throughout the welding period.

3. Welding Atmosphere: Welding shall be conducted in an inert gas which does not exceed 5 ppm oxygen, 15 ppm nitrogen, and 20 ppm moisture content (10 ppm moisture content in the case of components to contain lithium). Moisture and oxygen contents shall be continuously monitored during welding. Calibration of the oxygen analysis system shall be checked before and after each day of the welding operation by comparison with inert gas having a known impurity content. The response of the moisture analysis system shall be checked during welding by comparison with system performance recorded during chamber qualification.

4. Power Supply: Direct current, straight polarity power shall be used for welding.

5. Quality Control Specimens: Prior to the time that the first piece is welded and after the last piece is welded in each inert-gas environment, bead-on-plate bend specimens shall be prepared without filler wire addition. The material shall be taken from the parent material that is being welded, if possible. In any event, the parent material for the bead-on-plate runs shall be similar to the parent material in thickness and composition. And the width of the weld beads shall be approximately the same as those produced in the parent material test component weldments. The bead-on-plate bend specimens shall be 2 cm (0.75-inch) wide (minimum) and 5 cm (2-inches) long (minimum) with a longitudinal weld bead. These specimens shall be retained for subsequent chemical analysis should further investigation be desired.

6. Process Contamination Control: Precautions shall be taken during welding to avoid contamination of the weld metal by the tungsten electrode. The completed weldment shall remain in the inert gas environment until it has cooled below 150° C (300° F).

7. Repair Welding: Repair welding shall only be done upon approval of the Project Manager.

F. Quality Acceptance Requirements

1. Weld Defects: Weld deposits shall be smooth and uniform in appearance, have complete fusion, and blend into the base metal. The welded joints shall be free of the following defects as indicated by visual and radiographic examination and other applicable inspection methods:

    a. Cracks of any type or size in the weld metal and adjacent base metal

    b. Craters and cracks associated with craters

    c. Surface imperfections (holes, irregular bead)

    d. Overlap of weld metal on the base metal

    e. Undercutting along the edges of the weld or depression of the weld metal below the adjacent base metal

    f. Damage to the weld metal and adjacent base metal by gaseous contaminants

g. Incorrect weld profile and size

h. Lack of complete (100%) joint penetration in groove welds

i. Incomplete fusion between weld beads or between weld metal and base metal

j. Porosity in the weld metal

2. Groove-Weld Reinforcement:

a. Weld reinforcement on the face side of groove-weld joints shall not exceed the following:

Base Metal Thickness cm, (Inch)	Height of Reinforcement Maximum - mm,(Inch)
up to 1.3 (1/2)	1.6 (1/16)
over 1.3 (1/2)	2.4 (3/32)

b. Weld root reinforcement shall not be more than 25 percent of the tube inside diameter based on averaging of the diameters at 90° interpreted from radiographs.

3. Fillet Weld Contour: The face of a fillet weld shall be at approximately equal angles to the sections it joins, unless specifically noted otherwise on the drawing. The weld face may be slightly convex, flat, or slightly concave. Convex welds shall have a maximum convexity of 0.1-S plus 0.8-mm (0.03-inch) where S is the average length in mm (inch) of the two legs of the filled weld.

4. Tungsten Inclusion Limits by Radiography:

a. The largest dimension of any single radiographic indication of tungsten inclusions shall not exceed -

(1) 0.25-mm (0.010-inch) on material up to 2.5-mm (0.10-inch) thick

(2) Greater than 10% of the metal thickness or 1-mm (0.04-inch), whichever is less, in material 2.5-mm (0.10-inch) thick and above.

b. The spacing between adjacent tungsten inclusions shall not be less than three times the metal thickness.

c. No more than three inclusions in any 2.5-cm (1-inch) of weld length shall be allowed.

V. QUALITY ASSURANCE PROVISIONS

A. Weld Inspection Methods

All welded joints shall be inspected for conformance to the quality requirements listed in IV.F.

1. Visual Inspection: For weld quality, visual inspection shall be done using a 4X magnifying glass or better.

2. Radiographic Inspection:

   a. General Method - Radiographic procedures shall conform to ASTM E94-62T. Radiographic quality control procedures shall be those described in ASTM E142-64.

   b. Weld Surface Preparation - Accessible surfaces of welds shall be prepared as necessary so that valleys between beads, weld ripples, and other surface irregularities are blended so that radiographic contract due to surface condition cannot mask or be confused with that of any defect.

   c. Disposition of Radiographs - Radiographs of weldments shall be retained for future referral.

3. Ultrasonic Inspection: Weld joints shall be rejected when any indication produces an amplitude equal to or greater than the amplitude produced by the applicable calibration hole or notch.

4. Liquid Penetrant Inspection: This shall be performed per ASTM E165-65.

5. Helium Leak Check: Weld joints shall be rejected when results of a helium leak check indicates leak rates greater than 1×10^{-8} standard cc/sec.

B. Qualification of Welding Equipment and Procedure

1. Application: Before welding actual columbium or tantalum alloy parts and components, new welding equipment and inert gas chambers shall be qualified as described below:

   a. Chambers Designed for Refractory Metal Welding - Welding equipment that has not been used for welding columbium and tantalum alloys to this Specification for a period of one (1) month shall be requalified.

b. Field Welding Chambers - Welding equipment that has not been used for welding columbium and tantalum alloys to this specification for a period of three (3) months shall be requalified.

2. Gloves: For all GTA welding, new gloves shall be inspected for outgassing and surface bleeding by a chamber evacuation cycle to at least 1×10^{-4} torr and 50° C (120° F) for four hours. The chamber interior and glove surfaces shall be examined for evidence of contamination following the test. During this test, clean copper bars or sheet will be located in line of sight of the gloves at no more than one foot distance. Copper discoloration will be taken to result from glove outgassing, presumably sulfur, during pumpdown. This test may have to be repeated until the outgassing of the gloves has diminished or the gloves are rejected because of the outgassing or for other performance characteristics such as permeability. The gloves employed must not compromise the system qualification requirements. No gloves shall be used which show discoloration of the copper.

3. Chamber and Tooling: The weld chamber, gloves, attendant equipment, and atmosphere monitors shall be set up as for welding with glove port covers removed after backfilling.

   a. Chambers Designed for Refractory Metal Welding - The system shall be continuously operated for a minimum of five hours to observe the overall system response and atmosphere degradation rate. Weld specimens per paragraph V.B.6. shall be prepared at the end of the five hours operation. Before the five hour operation, the inert gas analysis for oxygen, water vapor, and nitrogen shall be available. During welding, oxygen and moisture contents shall be monitored per IV.E.3.

   b. Field Welding Chambers - The system shall be continuously operated for a minimum of two hours to observe the overall system response and atmosphere degradation rate. Weld specimens per paragraph V.B.6. shall be prepared at the end of the two hour operation.

4. Inert Gas Purity and Monitoring: Before the five hour or two hour operations described in V.B.3.a. or V.B.3.b., the inert gas analysis for oxygen, moisture, and nitrogen shall be available. During welding, oxygen and moisture contents shall be monitored per IV.E.3.

5. Response of Inert Gas Analysis System: Near the end of the chamber tests (V.B.3.a. or V.B.3.b.) and after welding the

specimens of paragraph V.B.6., approximately 20 ppm oxygen (80 ppm nitrogen), and 50 ppm water vapor impurities shall be suddenly introduced into the chamber to record the monitoring system response.

6. Evaluation Specimen Preparation: A full penetration fusion pass shall be made on two samples of the appropriate alloy during the equipment qualification. Welding procedures shall be in accordance with the requirementsof Section IV.E. The material thickness shall be approximately the same as the parts to be welded with a maximum thickness requirement of 3.2-mm (1/8-inch). Each specimen shall be 6.3-cm (2.5-inches) (minimum) in length and 2.5-cm (1.0-inch) (minimum) in width. The weld shall be centered within the specimen and parallel to the 6.3-cm (2.5-inch) side. Both specimens shall be welded at the end of the chamber test (V.B.3.a. or V.B.3.b.) but before intentional contamination.

7. Bend Tests: One weld specimen shall be used for bend tests as described below. These tests shall generally be performed at room temperature.

   One bend specimen shall be cut 6.3-cm (2.5-inches) long (minimum) by 2-cm (0.75-inch) wide (minimum). The weld axis shall be centered within the specimen and parallel to the long dimension. Any reinforcement on the root of the weld shall be removed before testing.

   Bend tests shall be performed with the specimens supported on an anvil having a 75 degree vee and a 28.6-mm (1-1/8-inch) span. Loading shall be applied using a 75 degree vee punch with a radius equal to the metal thickness being tested. Specimens shall be placed with the face of the weld down and centered under the wedge with the weld axis perpendicular to the bend axis. Specimens shall be bent 90 to 105 degrees at a uniform 5-mm (0.2-inch) per minute ram speed. After the first bend has been made, the specimen shall be examined for cracks, cut into halves, and each half bend tested. All three bend test specimens shall be examined for cracks at a magnification of 10X and shall exhibit no evidence of cracking.

8. Chemical Analyses: Chemical analyses of the parent metal and weld metal for oxygen, nitrogen, hydrogen, and carbon shall be made from the second weld specimen. Gas analyses shall be by vacuum fusion techniques, and the carbon shall be determined by the combustion method. The difference in concentration of any one of the constituents between the parent metal and weld metal (concentration in weld metal minus concentration in parent metal) shall not exceed the limits shown in the following table:

Element	Increase in Concentration* Cb-alloys	Ta-alloys
Carbon	15 ppm	10 ppm
Oxygen	30 ppm	20 ppm
Nitrogen	30 ppm	20 ppm
Hydrogen	2 ppm	2 ppm

9. Qualification Report: Full records of these qualification tests shall be retained for future referral.

C. Rejection of Weldments

Weldments and components not conforming to this specification shall be rejected.

D. Reports

A report shall be submitted with the finished parts and shall include the information, but not necessarily the format, of Exhibit I. This report shall be prepared for each welding cycle. A sketch of the welding arrangement shall be included in the report.

These reports shall be retained for future referral should subsequent weld evaluation be desired.

*The values given for permissible increase in concentration of interstitials include an allowance for variations in analyses due to the precision of the analytical method.

EXHIBIT I

PROCESS CONTROL RECORD

Date	Subject
Contract No.	Gas Tungsten-Arc Welding Columbium, Tantalum, and Their Alloys

Part Name	Drawing No.	Weld No.

Welding Machine, Manufacturer, and Model:

Welding Chamber:

Pumping System:

Tungsten Electrodes:

Filler Metal:

Inert Gas:

Fixturing:

Welding Data:

Voltage

Amperage

Travel Speed

Other Pertinent Data

Sketch of Welding Arrangement: (below)

NASA-LEWIS SPECIFICATION NO. RM-3

ELECTRON BEAM WELDING OF

COLUMBIUM, TANTALUM, AND THEIR ALLOYS

JUNE 1971

I. SCOPE

This Specification defines procedure requirements, process substantiation, and quality requirements for electron beam welding of columbium, tantalum, and their alloys. It may also be applied to electron beam welding of other metals and alloys which are readily contaminated during welding.

II. APPLICABLE DOCUMENTS

AWS A3.0-69	Terms and Definitions
AWS A2.2-68	Standard Welding Symbols
NASA-Lewis Specification No. RM-1	Chemical Cleaning of Columbium, Tantalum, and Their Alloys
NASA-Lewis Specification No. RM-5	Postheating Cb-1Zr and T-111 (Ta-8W-2Hf) Weldments
AWS A2.2-69	Nondestructive Testing Symbols
ASTM E165-65	Liquid Penetrant Inspection
ASTM E94-62T	Tentative Recommended Practice for Radiographic Testing
ASTM E142-64	Controlling Quality of Radiographic Testing

III. DEFINITIONS

Electron Beam Welding: A fusion welding process wherein coalescence is produced by the heat obtained from a concentrated beam composed primarily of high velocity electrons impinging upon the surfaces to be joined.

IV. REQUIREMENTS

A. Equipment

1. Welding Machine: An electron beam welding machine shall be used in which welding is accomplished inside a vacuum-tight

chamber. The electron beam machine shall be capable of operation at accelerating voltages of 25 KV or higher.

2. Welding Chamber: The welding chamber shall be capable of evacuation to 1×10^{-5} torr. It shall have a maximum total rate of pressure rise of 1×10^{-2} torr per hour starting at a chamber pressure of 1×10^{-4} torr.

3. Electron Beam Alignment: The machine shall be equipped with proper visual or electronic aids to align and maintain the correct relationship between the electron beam and the work during the welding operation.

4. Focus of the Electron Beam: The beam shall be focused on a refractory metal block (copper blocks shall not be used).

5. Welding Machine Operator: Welding machine operators who perform electron beam welding to this specification shall either have been trained by a bona fide manufacturer of electron beam equipment or shall operate under direction of such a trained operator.

6. Fixture Materials: Fixtures and other holding devices used during welding shall be made from materials that do not corrode in air. Fixture components that contact refractory metal weldments shall be made of molybdenum, tantalum, columbium, tungsten, or their alloys. Under no circumstances shall a refractory metal alloy component to be welded contact any nonrefractory metal during welding.

7. Fixture Cleanliness: Fixtures shall be clean and free of surface contaminants such as grease, oil, and dirt of any kind.

B. Cleaning and Handling

1. Cleaning: The parts to be joined shall be chemically cleaned in accordance with Specification No. RM-1, "Chemical Cleaning of Columbium, Tantalum, and Their Alloys."

2. Handling: Cleaned components, parts, and assemblies shall be handled with clean lint-free gloves.

C. Welding Procedures

1. Qualification: Welding shall be done using equipment and procedures which have been qualified per paragraph V.B.

2. Hardware: Welding procedures shall be established prior to welding actual hardware.

3. Chamber Pressure: Prior to welding, the chamber shall be evacuated to 5×10^{-5} torr or lower. The pressure during welding shall not rise above 5×10^{-4} torr.

4. Cooling: The completed weldment shall be left in the chamber for a sufficient length of time for it to cool to less than 150^{o} C (300^{o} F).

5. Repair Welding: Repair welding shall only be done upon approval of the Project Manager.

D. Quality Acceptance Requirements

Welded joints shall be free of the following defects established by any and all applicable methods of inspection.

1. Cracks in the weld and adjacent base metal
2. Crater cracks
3. Surface imperfections (holes, irregular bead)
4. Undercutting along the edges of the weld
5. Depression of the weld metal below adjacent base metal (concave surface at face of weld)
6. Damage to weld metal and adjacent base metal by contamination from foreign materials and the ambient atmosphere
7. Incomplete fusion
8. Partial joint penetration of groove welds
9. Overlap of weld metal on base metal
10. Shrinkage cavities
11. Porosity in weld metal

V. QUALITY ASSURANCE PROVISIONS

A. Weld Inspection Methods

All welded joints shall be inspected for conformance to quality requirements listed in IV.D.

1. Visual Inspection: Visual inspection for weld quality shall be done using a magnifying glass of 4X magnification or better.

2. Ultrasonic Inspection: Electron beam welded joints shall be rejected when any indication produces an amplitude equal to or greater than the amplitude produced by the applicable calibration hole or notch.

3. Liquid Penetrant Inspection: This shall be performed per ASTM E165-65.

4. Helium Leak Check: Welds shall be rejected when results of a helium leak check indicates leak rates greater than 1×10^{-8} standard cc/sec.

5. Radiographic Inspection: Inspection shall reveal no cracks or voids using quality control procedures conforming to ASTM E94-62T.

B. Qualification of Welding Equipment and Procedure

1. Electron Beam Vacuum Chamber: Prior to welding the first piece, the total rate of pressure rise in the welding chamber shall be determined per IV.A.2.

2. Welding of Sheet Coupon for Bend Testing: Prior to welding actual components, a full penetration bead-on-plate weld shall be made in a sheet coupon of the same alloy as production parts. The coupon shall be about the same thickness as the production weld joint but with a minimum of 0.8-mm (.03-inch) and a maximum of 3.2-mm (.125-inch) and a length of about 15-cm (6-inches).

3. Cutting of Coupon: Four strips 1.3-cm (1/2-inch) wide x 3.2-cm (1-1/4-inch) long shall be cut from the coupon with the weld bead parallel to the length and centered.

4. Procedure for Bend Testing: Three of the four welded strips shall be bend tested transverse to the length using a 75° V-block and mating V-punch with a bend radius equal to the metal thickness. Weld drop-thru shall be removed before testing. Two coupons shall be bent with weld face in tension and a third coupon with the weld root in tension.

5. Bend Test Requirements: All test specimens shall be bent 90° to 105° at a uniform 5-mm (0.2-inch) per minute ram speed. The bent material shall be examined for cracks at 4X magnification.

6. Storage of Extra Welded Strip: A fourth welded strip shall accompany the production welded pieces. This strip shall be stored with the process control records for future reference.

7. Requirements for Partial Penetration Welds: If a partial penetration weld is required, such as in an end-cap-to-cylinder weld, metallographic sections shall be taken longitudinal and transverse to the welding direction. Examination shall be made for root defects such as voids, pores, or cracks. If such defects are present, the welding parameters shall be adjusted in order to avoid these defects.

C. Rejection of Weldments

Weldments and components not conforming to this specification shall be rejected.

D. Reports

Reports shall be submitted with finished parts giving all vacuum measurement, welding data, preheating, postheating, and welding equipment and procedure qualification test data. Inspection reports shall also be furnished as required by other appropriate specifications. All reports, X-rays, charts, etc., shall reference the appropriate drawing number, part number, and weld number. This report for each welding cycle shall include the information listed in Exhibit I and other pertinent information.

These reports shall be retained for future referral should subsequent weld evaluation be desired.

EXHIBIT I

PROCESS CONTROL RECORD

Date	Subject	
Contract No.	Electron Beam Welding of Columbium, Tantalum, and Their Alloys	
Part Name	Drawing No.	Weld No.

Welding Machine:

Manufacturer

Model

Welding Chamber:

Pumping System:

Electron Gun:

Fixturing:

Welding Controls:

Welding Data:

Voltage

Amperage

Travel Speed

Beam Motion

Chamber Pressure

Other Pertinent Data

Sketch of Welding Arrangement: (below)

NASA-LEWIS SPECIFICATION NO. RM-4

RESISTANCE-SPOT WELDING OF REFRACTORY METAL FOIL TO REFRACTORY METAL COMPONENTS

JUNE 1971

I. SCOPE

This specification covers the procedures to be followed for all resistance spot welding for attaching refractory metal foil to refractory metal piping and other components.

II. APPLICABLE DOCUMENTS

AWS A3.0-69	Terms and Definitions
AWS A2.0-68	Standard Welding Symbols
AWS C1.1-66	Recommended Practices for Resistance Welding
NASA-Lewis Specification No. RM-1	Chemical Cleaning of Columbium, Tantalum, and Their Alloys
NASA-Lewis Specification No. RM-5	Postheating Cb-1Zr and T-111 (Ta-8W-2Hf) Weldments

III. DEFINITION

Resistance Spot Welding: A resistance-welding process wherein coalescence at the faying surfaces is produced in one spot by the heat obtained from the resistance to electric current through the work parts held together under pressure by electrodes.

IV. REQUIREMENTS

A. Equipment

1. Welding Machine: A capacitance discharge spot welding machine is required.

2. Electrodes: The resistance spot welding machine shall be equipped with molybdenum electrodes with broad tips, 3.2-mm (1/8-inch) diameter.

3. Argon Shielding: An argon supply tube shall be attached to the weld gun with the end of the tube located 1.3-cm (1/2-inch) from the electrode tip. Argon shall be supplied through this tube to blanket the spot weld area at all times when spot welds are made.

4. Power Setting: Power/energy setting shall not exceed 200 watt-seconds and shall be determined by making sample welds on refractory metal specimens of the same material and thickness prior to making the welds on the actual system or components.

5. Ground Clamp: Ground connections shall be made to a nonrefractory metal portion of the system (e.g., stainless steel portion of bimetallic joint) when possible. If a suitable stainless steel location is not available, then a refractory metal (columbium, tantalum, tungsten, molybdenum, or their alloys) clamp must be attached to an appropriate location on the system and the ground clamp affixed to the refractory metal clamp. Under no circumstances shall an electrical connection for spot welding be made directly between a refractory metal component of the system and a nonrefractory metal ground clamp.

6. Fixturing: Nonrefractory metal fixturing shall not be permitted to contact the refractory metals being welded during the spot welding process.

B. Cleaning and Handling

1. Cleaning: The refractory metal component and the refractory metal foil that are to be spot welded shall be cleaned per NASA-Lewis Specification RM-1.

2. Handling: Cleaned components, parts, and assemblies shall be handled with clean, lint-free gloves.

C. Welding Procedure

The power/energy setting shall be adjusted and the molybdenum electrodes designed to produce argon-shielded resistance-spot welds. These procedures shall be established (per V.B.) prior to welding actual hardware.

D. Quality Acceptance Requirements

The spot welds shall show no cracks, voids, or expelled metal.

V. QUALITY ASSURANCE PROVISIONS

A. Weld Inspection Method

All spot welds shall be inspected visually using a 4X magnification or better.

B. Qualification of Welding Procedure

Prior to spot welding the foil to the component, a single spot weld shall be made in sheet coupons of the same alloy in about the same thicknesses. Peel tests shall be made per AWS C1.1-66 to demonstrate that sufficient penetration has been achieved. Metal from the foil must be torn out in its entirety (as a button) in order to demonstrate adequate weld quality.

C. Reports

A process control sheet (Exhibit I) shall be completed by the operator for each series of spot welds applied to a single refractory metal component or a section of system piping between components. These reports shall be retained as a part of the quality assurance records for the system being constructed.

EXHIBIT I

PROCESS CONTROL RECORD

Subject: Resistance-Spot Welding Refractory Metal Foil to Refractory Metal Components

Contract No.:

Installation Drawing No.:

Name of Component or
Description of Pipe:

Model and Serial No. of Spot Welding Machine:

Type of Electrode Used:

Argon Cover Gas On:

Type and Thickness of Material Welded:

Sample Weld Made:

Watt-Second Setting Used:

Location of Ground Connection:

Remarks:

NASA-LEWIS SPECIFICATION NO. RM-5

POSTHEATING OF Cb-1Zr AND T-111 (Ta-8W-2Hf) WELDMENTS

JUNE 1971

I. SCOPE

High temperature aging reactions, resulting in weld embrittlement, make it necessary to postheat* all Cb-1Zr weldments and T-111 (Ta-8W-2Hf) weldments which will be exposed to liquid alkali metals. This specification establishes the procedures, process substantiation, and quality requirements for postheating all Cb-1Zr and T-111 gas tungsten-arc and electron beam weldments.

II. APPLICABLE DOCUMENTS

AWS A3.0-69	Terms and Definitions
NASA-Lewis Specification No. RM-1	Chemical Cleaning of Columbium, Tantalum, and Their Alloys

III. REQUIREMENTS

A. Equipment

1. Qualification: Vacuum annealing shall be performed using equipment which has been qualified in accordance with Section IV.

2. Heat Sources: Weldments may be annealed individually by application of local heat sources in a vacuum environment or by vacuum furnace annealing of welded assemblies.

3. Local Heating: The heater shall have only refractory metals in the hot zone. Dense vitrified alumina (99.2% purity) shall be used as the heater electrical insulation in the cold zone of the furnace.

4. Vacuum Furnaces: Vacuum furnaces shall be radiant-heated, cold-wall type with refractory metal heater elements and primary heat shields. Permanent hearth materials and support fixtures shall be constructed from refractory metals.

B. Cleaning

Weldments or assemblies whose surfaces have been contaminated subsequent to welding shall be cleaned in accordance with NASA-Lewis Specification No. RM-1, "Chemical Cleaning of Columbium, Tantalum, and Their Alloys."

*Postheating: Any heating of a weldment immediately after welding.

C. Annealing Procedures

1. Protective Wrappers: Materials to be annealed shall be enclosed in a chemically-cleaned tantalum, columbium, or Cb-1Zr alloy container or wrapped with at least one overlapping layer of clean tantalum, columbium, or Cb-1Zr alloy foil 0.05-mm (0.002-inch) thick or greater. In local annealing, the above wrap is necessary only over the heated portion of the assembly.

2. Vacuum: For either local or vacuum furnace annealing, the pressure shall be maintained at 1×10^{-5} torr or less when the part temperature is above 260° C (500° F) and a maximum of 1×10^{-4} torr during the time that the part is being brought to temperature. The part shall not be exposed to air until it has cooled below 150° C (300° F).

3. Annealing Cycle: The weldments shall be postheated as follows:

   Cb alloys: 1200° C $\pm$ 13° C - 1 hour (2200° F $\pm$ 25° F - 1 hour)

   Ta alloys: 1315° C $\pm$ 13° C - 1 hour (2400° F $\pm$ 25° F - 1 hour)

   The heating rate for these anneals shall not exceed 650° C (1200° F) per hour.

4. Temperature Measurement: The temperature of the weld joint shall be measured by at least one W-Re type thermocouple held in mechanical contact with the weld joint region. Pt/Pt-Rh thermocouples may be used if the thermocouple junction is separated from the weldment by at least one wrap of protective refractory metal foil. Intimate contact is necessary at the thermocouple-foil-weldment interfaces to assure true weldment surface temperature measurement. When numerous weldments are annealed as an assembly, at least two W-Re thermocouples shall be attached to weldments representing the thinnest and heaviest weld cross-sections of the assembly. Additional thermocouples shall be attached to the assembly as required to provide uniform temperature distribution; i.e., normally, a point closest to the heater and at the center of the assembly.

5. Fixturing: Assemblies shall be fixtured or positioned in the furnace to minimize distortion during the annealing cycle. All fixtures, supports, or furnace members which contact the parts or assemblies during the annealing cycle shall be made from refractory metals.

6. Quality Control Specimens: At least one control specimen of the parent alloy shall accompany the welded components or assemblies during postheating in vacuum. This specimen shall weigh 10 grams or more, with a thickness representative of the components being annealed. After chemical cleaning, the specimen may be enclosed under the protective foil wrap described in III.C.1., or it may be wrapped with foil in a manner equivalent to the parts or assemblies.

   The above specimens shall be permanently marked with their material control number and shall be submitted with the finished parts. Specimens shall be tagged with the drawing number, part number (weld number when localized annealing is used) and serial number of each part or assembly represented by the specimens.

   These quality control specimens shall be retained for subsequent chemical analysis should an investigation of vacuum environment contamination be desired.

IV. QUALITY ASSURANCE PROVISIONS

A. Qualification of Annealing Equipment and Procedures

Before annealing welded joints or components, new annealing procedures and equipment shall be qualified or described below to insure that weldments are not contaminated nor embrittled during heat treatment. Once qualified, the furnace can lose its qualification status for any one of the following reasons:

1. Prolonged Disuse: The furnace has not been used for annealing columbium, molybdenum, tungsten, tantalum, or their alloys to this specification for a period of three months.

2. Nonrefractory Metal Usage: Metals other than columbium, tantalum, molybdenum, tungsten, or their alloys are annealed in this furnace.

3. Specimen Contamination: Analysis of qualification specimens (IV.B.) indicate furnace contamination.

B. Qualification Specimens

Two specimens of either Cb-1Zr or T-111 alloy, each weighing 10 grams or more, shall be mechanically sheared from sheet having a thickness representative of the components to be annealed with a maximum thickness of 1.6-mm (1/16-inch). Both specimens shall be chemically cleaned per NASA-Lewis Specification No. RM-1.

C. Vacuum Annealing

One qualification specimen shall be wrapped in protective foil per III.C.1. and shall be annealed using the annealing cycle, procedures, and equipment in accordance with the requirements of Section III.

D. Reference Specimen

The other specimen shall be retained unannealed to be analyzed with the annealed specimen. The analysis of the unannealed specimen shall be the reference standard to which the analysis of the annealed specimen is compared for contamination qualification.

E. Chemical Analyses

Chemical analyses of both the annealed and unannealed specimens shall be made for oxygen, nitrogen, hydrogen, and carbon. Gas analyses shall be by vacuum fusion techniques, and carbon shall be determined by the combustion method. The difference in concentration of any one of the constituents between the annealed and unannealed specimen (concentration in annealed specimen minus concentration in the unannealed specimen) shall not exceed the limits shown in the following table:

	Increase in Concentration*	
Element	Cb-1Zr	T-111
Carbon	15 ppm	10 ppm
Oxygen	30 ppm	20 ppm
Nitrogen	30 ppm	20 ppm
Hydrogen	2 ppm	2 ppm

F. Rejection

Weldments and components whose annealing treatment has not conformed to this specification shall be rejected.

G. Reports

A report shall be submitted with the finished parts and shall include the information, but not necessarily the format of Exhibit I. This report shall be prepared for each postheating treatment. This report shall be retained for future referral should a subsequent evaluation of vacuum environment contamination be desired.

*The values given for permissible increase in concentration of interstitials include an allowance for variations in analyses due to the precision of the analytical method.

EXHIBIT I

PROCESS CONTROL RECORD

Subject: Postheating Cb-1Zr and T-111 (Ta-8W-2Hf) Weldments

Contract No.:

A. Equipment

1. Certification Date
2. Manufacturer and Serial Number of Furnace
3. Manufacturer and Serial Number of Temperature Readout
4. Manufacturer and Serial Number of Vacuum Monitoring Equipment

B. Records

The time/temperature/vacuum records are to be attached to this record.

REFERENCES

1. Frank, R. G.; Miketta, D. N.; Kearns, W. H.; Young, W. R.; and Hand, R. B.: Material and Process Specifications for Refractory Alloy and Alkali Metals. Rep. R66SD3007, General Electric Co. (NASA CR-88711), Dec. 13, 1965.

2. Anon.: Recent Advances in Refractory Alloys for Space Power Systems. NASA SP-245, 1970.

3. Bond, James A.: The Design of Components for an Advanced Rankine Cycle Test Facility. Presented at the Fifth Intersociety Energy Conversion Engineering Conference, Las Vegas, Nev., Sept. 21-25, 1970.

4. Mendelson, Irwin: Design and Fabrication of a Brayton Cycle Solar Heat Receiver. NASA CR-72872.